REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
20-01-1998	Paper	

4. TITLE AND SUBTITLE

The Far-Reaching Nature of Microtube Technology

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER
2306

5e. TASK NUMBER
M1B3

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/PRS
5 Pollux Drive
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8. PERFORMING ORGANIZATION REPORT

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/PRS
5 Pollux Drive
Edwards AFB CA 93524-7048

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S NUMBER(S)
AFRL-PR-ED-TP-1998-036

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
					Wesley Hoffman
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified	A		19b. TELEPHONE NUMBER *(include area code)* (661) 275-5768

98-036

ORIGINAL ARTICLE

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The far-reaching nature of microtube technology

Received: 29 January 1998 / Accepted: 10 March 1998

Abstract Microtubes have potential applications in numerous diverse fields. They are very small diameter tubes (in the nanometer and micron range) with high aspect ratios that can be made from practically any material. In contrast, to other technologies, tubes larger than 5 microns in diameter can be made from any material with any combination of cross-sectional and axial shapes desired. The significance of microtube technology is that it provides solutions to many materials problems as well as the opportunity to miniaturize numerous components and devices that are currently in existence. In addition, components that have to date been impossible to produce can be fabricated using this technology.

Key words Microtubes · Microtubules · Microchannels · Microsprings · Microbellows

Introduction

In recent years there has been tremendous interest in miniaturization due to the high payoff involved. The most graphic example that can be cited has occurred in the electronics industry which only 50 years ago relied upon the vacuum tube. The advent of the transitor in 1947 started a revolution in miniaturization that was inconceivable at the time of its invention, and not fully recognized even many years later.

Miniaturization, resulting in the possibility for hundreds of millions of transistors to occupy the volume of a vacuum tube or the first transistor, was not the only result. The subsequent spin-off developments in allied areas, such as integrated circuits and the microprocessor, have spawned entirely new fields of technology. It is quite likely that other areas are now poised for revolutionary developments that parallel those that have occurred in the electronics industry since the advent of the first transistor.

These areas would include microelectromechanical systems (MEMS) and closely related fields, such as micro-fluidics and micro-optical systems. Currently, these technologies involve micro-machining on a silicon chip to produce numerous types of devices, such as, sensors, detectors, gears, engines, actuators, valves, pumps, motors, and mirrors on the micron scale. The first commercial product to arise from MEMS was the accelerometer manufactured as a sensor for air-bag actuation. On the market today, there are also micro-fluidic devices, mechanical resonators, biosensors for glucose, and disposable blood pressure sensors that are inserted into the body.

The vast majority of microsystems are made almost exclusively on planar surfaces using technology developed to fabricate integrated circuits. That is, the fabrication of these devices takes place on a wafer and the device is formed layer-by-layer with standard cleanroom techniques that include e-beam or photolithography, thin-film deposition, and wet or dry etching. There are several variations of this classic technology, which include imprint lithography involving compression molding [1], laser micromachining [2], electrochemical micro-machining [3], and very limited application of deposition using a scanning tunneling microscope [4]. However, the variation of conventional chip technology most employed is the LIGA process [5]. This process was developed specifically for MEMS-type applications and is able to construct and metallize high-aspect-ratio microfeatures. This is done by applying and exposing to synchrotron radiation a very thick X-ray sensitive plastic. Features of 300 microns in height and with aspect ratios of 300 to 1 can be fabricated by this technique making truly 3-dimensional objects. However, like the conventional technique, all these variations use a layered approach starting on a flat surface.

Although there have been numerous and very innovative successes using these silicon wafer-based technolo-

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gies, there are some disadvantages. Since it requires the building-up of many layers of different materials as well as surface and bulk micro-machining there are some very difficult material science problems to solve. These include differential etching and laying down one material without damaging a previous layer. In addition, there are the problems associated with interconnecting layers in a chip with different functions. An example of this would be a micro-fluidic device in which there are both fluidic and electronic functions. Clearly, there are numerous materials issues central to this technology.

In addition to these processing problems, there are other limitations inherently associated with conventional lithographic techniques that are based on planar silicon. For example, in some applications such as those that involve surface tension in fluidics, it is important to have a circular cross-section. However, it is impossible to make a perfectly round tube or channel on a chip with current technology. Instead channels are made by etching a trench and then covering the trench with a plate. This process can only produce angled channels such as those with a square, rectangular, or triangular cross-section. Thus, we would heartily agree with Wise and Najafi in their review of micro-fabrication technology [6] when they stated "The planar nature of silicon technology is a major limitation for many future systems, including micro-valves and pumps".

In the literature, there are at least two technologies in addition to microtubes that remove microfabriation from the flatland of the wafer. One makes use of contact printing while the other utilizes laser-assisted chemical vapor deposition (LCVD). Microcontact printing and other forms of "soft lithography" are very novel techniques developed by Whitesides' group at Harvard [7–8]. With these techniques submicron features can be fabricated on flat or curved substrates. In microcontact printing this is accomplished by "inking" a stamp with an alkanethiol which is then brought into contact with a gold-coated surface, for example, by rolling the curved surface over the stamp. The gold is then etched where there is no self-assembled monolayer of alkanethiolate. Features as small as 200 nm can be formed with this technique. However, it should be mentioned that the microstructures produced by this technique are the same as those produced by standard techniques with the exception that the starting surface need not be flat.

The next step away from the standard planar silicon technology is the LCVD process [9–10] which is able to "write in space" producing truly three-dimensional microsystems. With this process two intersecting laser beams are brought to focus in a very small volume in a vacuum chamber. The surface of the substrate on which deposition is to occur is brought to the focal point of the lasers. The power to the lasers is adjusted so that deposition from the gas phase occurs only at the intersection of the beams. As deposition occurs on the substrate surface, it is pulled away from the focal point. Under computer control, the substrate can be manipulated so that complex free-standing three-dimensional microstructures can be fabricated.

In addition to LCVD, only microtube technology offers the possibility of truly 3-dimensional non-planar microsystems. However, in contrast to LCVD technology, microtube technology also offers the ability to make micro-devices out of practically any material since the technology is not limited by the availability of CVD precursor materials. In addition, microtube technology provides the opportunity to make tubing of any configuration in order to miniaturize systems, connect components, and fabricate components or systems that are currently not possible to produce.

Commercially, tubing is extruded, drawn or pultruded, which limits the types of materials that can be used for ultra-small tubes as well as their ultimate internal diameters. In addition it is not currently possible to control wall thicknesses to a fraction of a micron with these techniques. Using conventional techniques, ceramic tubes are currently available only a small as 1 mm ID, copper tubing can be obtained as small as 0.05 mm ID, polyimide tubing is fabricated as small as 80 microns ID and quartz tubing is drawn down as small as 2 microns ID. This means that quartz is the only tubing commercially available that is less than 10 microns in internal diameter. This quartz tubing is used principally for chromatographic applications.

There are, however, other sources of small tubing which are presently at various stages of research and development. Several groups have been using lipids as templates [11–13] in order to fabricate submicron diameter tubing. These tubes are made by using an electroless deposition technique to metallize a tubular lipid structure formed from a Langmuir-Blodgett film. Lipid templated tubes are very uniform in diameter, which is fixed at ~0.5 microns by the lipid structure. Lengths of 100 microns have been obtained with this technique which is extremely expensive due to the cost of the raw materials.

Other groups are making sub-micron diameter tubules using a membrane-based synthetic approach. This method involves the deposition of the desired tubule material within the cylindrical pores of a nanoporous membrane. Commercial "track-etch" polymeric membranes as well as anodic aluminum oxide films have been used as the porous substrate. The aluminum oxide, which is electrochemically etched, has been the preferred substrate because pores of uniform diameter can be made from 5-1000 nm. From the liquid phase Martin [14–16] polymerized electrically conductive polymers and electrochemically deposited metal in the pore structure of the membrane. Kyotani et al. [17–18] have deposited pyrolytic carbon inside the pores of the same type of alumina substrate. After the inside walls of the porous membrane are covered to the desired thickness, the porous membrane is dissolved leaving the tubules. As with the lipid process, all the tubules formed by this process in a single membrane are uniform in diameter, length and thickness. But in contrast to the lipid process the diameter of the tubules can be varied by the extent of oxidation of the aluminum substrate. Although diameters can be varied with this process, it should be clear that these tubules are lim-

ited in length to the thickness of the porous membrane. In addition, the wall thickness is also limited in that the sum of the inside tubule diameter and two times the wall thickness are equal to the starting pore diameter.

Using a sol-gel method, tubules can be made in about the same diameter range as with the membrane approach. By hydrolyzing tetraethlyorthosilicate at room temperature in a mixture of ethanol, ammonia, water and tartaric acid, Nakamura and Matsui [19] were able to make silica tubes with both square and round interiors. The tubules produced by this technique had lengths up to 300 microns while the interior diameter of the tubes ranged from 0.02 to 0.8 microns.

On an even smaller scale, nanotubules are fabricated using a number of very different techniques. The most well known tube in this category is the carbon "buckytube" that is a cousin of the C_{60} buckyball [20-22]. Since carbon nanotubes were first observed as a by-product in C_{60} production, the method of C_{60} formation using an arc discharge plasma was modified to enhance nanotube production. The process produced tubules with the internal diameter in the range 1–30 nanometers. These tubules are also limited in length to about 20 microns. Similar nanotubes of BN [23], B_3C, and BC_2N [24] have been made by a very similar arc discharge process.

An alternative technique to manufacture carbon nanotubes has been known to the carbon community for decades through the work of Bacon, Baker and others [25–26]. The process produces a hollow catalytic carbon fiber through the pyrolysis of a hydrocarbon gas over a catalyst particle. The fibers, which vary in diameter from 1 nm to 0.1 micron and in lengths up to centimeters, can either be grown hollow or the amorphous center can be removed by catalytic oxidation after a fiber is formed.

Other nanoscale tubules with diameters both slightly larger and smaller than buckytubes have been made from bacteria and components of cytoskeletons as well as by direct chemical syntheses. Chow and others [27] isolated and purified nanoscale protein tubules called rhapidosomes from the bacterium *Aquaspirillum itersonii.* After the rhapidosomes were metallized by electroless deposition, metal tubules of approximately 17 nm in diameter and 400 nm in length were produced. Using a similar metallization technique, metal tubes have been fabricated [28] with inner diameters of 25 nm using biological microtubules as templates. These microtubules, which are protein filaments with outer diameters of 25 nm and lengths measured in microns, are components of the cytoskeletons of eukaryotic cells. In contrast to tubules produced from biological templates, the tubules produced by direct chemical synthesis involve using the technique of molecular self-assembly. Some of the nanotubules that fall into this category are those made from cyclic peptides [29] and cyclodextrins [30]. Cyclic peptide nanotubules have an internal diameter of 0.8 nm and can be made several microns in length. Other self-assembled nanotubules with internal diameters from 0.45 to 0.85 nm have been synthesized from cyclodextrins [30–31] with lengths in the tens of nanometers.

Although it is clear that individual nanotubules are currently useful for certain applications, such as, encapsulation or reinforcement, it is not obvious how nanotubules can be observed and manipulated for use in devices other than with scanning probe microscopes [32]. Until this problem is solved, the future of individual nanotubes in devices is uncertain. However, this problem can be circumvented if the nanotubules are part of a larger body.

If oriented groups or arrays of sub-micron to micron diameter tubes or channels are desired, there are at last three means available to make them. On a two-dimensional plane, channels that range in size from tens to hundreds of microns in width and depth have been fabricated [33–34] on the surface of silicon wafers using standard micro-photo-lithographic techniques. For smaller tubes or channels a technique [35] has recently been developed to draw down bundles of tubes to form an array. This process produces a hexagonal array of glass tubes each as small as 33 nm in diameter. This translates to a density of 3×10^{10} channels per square centimeter. Even smaller regular arrays of channels can be synthesized by a liquid crystal template mechanism [36]. In this process, aluminumsilicate gels are calcined in the presence of surfactants to produce channels with diameters of 2–10 nanometers.

It is quite apparent from this brief and incomplete review, that a number of very novel and innovative approaches have been used to make microsystems as well as tubes and channels with diameters in the range of nanometers to microns. It is the intention of this communication to very briefly summarize some of the work at the Air Force Research Laboratory (AFRL) in these areas.

Relation to previous work

In order to ascertain previous work in the field, a Chemical Abstracts as well as a Current Contents search was performed using the key words: tubes, tubules, microtubes, and microtubules. The search revealed the work described in the introduction as well as related work which consisted essentially of minor variations of the work described. Except for self-assembled tubules, microtube technology is able to produce tubes in the size range of all the other techniques cited. In contrast to all the other techniques, our technology is able to produce tubes with a great diversity of axial and cross-sectional geometries as well as very accurately controlled wall thickness and composition. In addition, the Air Force Research Lab technology is able to produce tubes with a much wider variety of materials than any other known process.

Experimental

The technique used to fabricate microtubes and microtube devices is less complex than some of those described above. However, this process offers numerous advantages that will be detailed below.

Like many of the techniques described above, the AFRL Lab approach employs a fugitive process with a sacrificial mandrel, which in this case is a fiber. By a proper choice of fiber, coating, deposition method, and mandrel removal method, tubes of practically any composition can be fabricated. Obviously, to make precision tubes of high quality, a great deal of material science is involved.

Results and discussion

In contrast to tubing currently available on the market and the sub-micron laboratory scale tubing mentioned above, microtubes can be made from practically any material with precisely-controlled composition, diameter, and wall thickness in a great range of lengths. There is no upper diameter limit, and with practically any material, internal diameters of less than 5 microns are possible. In addition, for materials that can survive temperatures greater than 400° C, tubes can be made as small as 5 nanometers using the same process.

To date, tubes have been made from metals (copper, nickel, aluminum, gold, platinum, silver) ceramics (silicon carbide, carbon, silicon nitride, sapphire), glasses (silica), polymers (Teflon), alloys (stainless steel) and layered combinations (carbon/nickel, silver/sapphire) in sizes from 0.5–410 microns. Some scanning electron microscope (SEM) micrographs of these tubes can be seen in Fig. 1.

Since the process does not involve pultrusion, extrusion or drawing but rather a very simple fugitive tube forming process, cross-sectional shapes as well as wall thickness can be very accurately controlled to a fraction of a micron. This is not possible with any of the approaches cited above. A myriad of cross-sectional shapes have already been made as can be seen in Fig. 2. These micrographs should be sufficient to demonstrate that practically any cross-sectional shape imagined can be fabricated. As seen in Fig. 2, the wall thickness of the tubes can be held very uniform around the tube. It is also possible to accurately control the wall thickness along the length of the individual tubes as well as among the tubes in a batch or a continuous process. In additional to the possibility of cross-sectional tube shapes, using a fugitive process also allows the fabrication of tubes with practically any axial geometry as will be shown below.

The maximum length that these tubes can be made has yet to be determined because it depends on many variables, such as, type of tube material, composition of sacrificial tube forming material, degree of porosity in the wall, etc. It is possible that with a porous wall there is no limitation in length. For a non-porous wall the maximum length would probably be in the meter range with there being a direct relationship between the tube ID and the maximum possible length. However, for most applications conceived to date, the length need only be on the order of a few centimeters. If one does a quick calculation it is apparent that even "short" tubes have a tremendous aspect ratio. For instance, a 10 micron ID tube 1 inch long has an aspect ratio of 2500.

With microtube technology, there is virtually no limitation in wall thickness. To date, free-standing tubes have been made with wall thickness as small as 0.01 microns

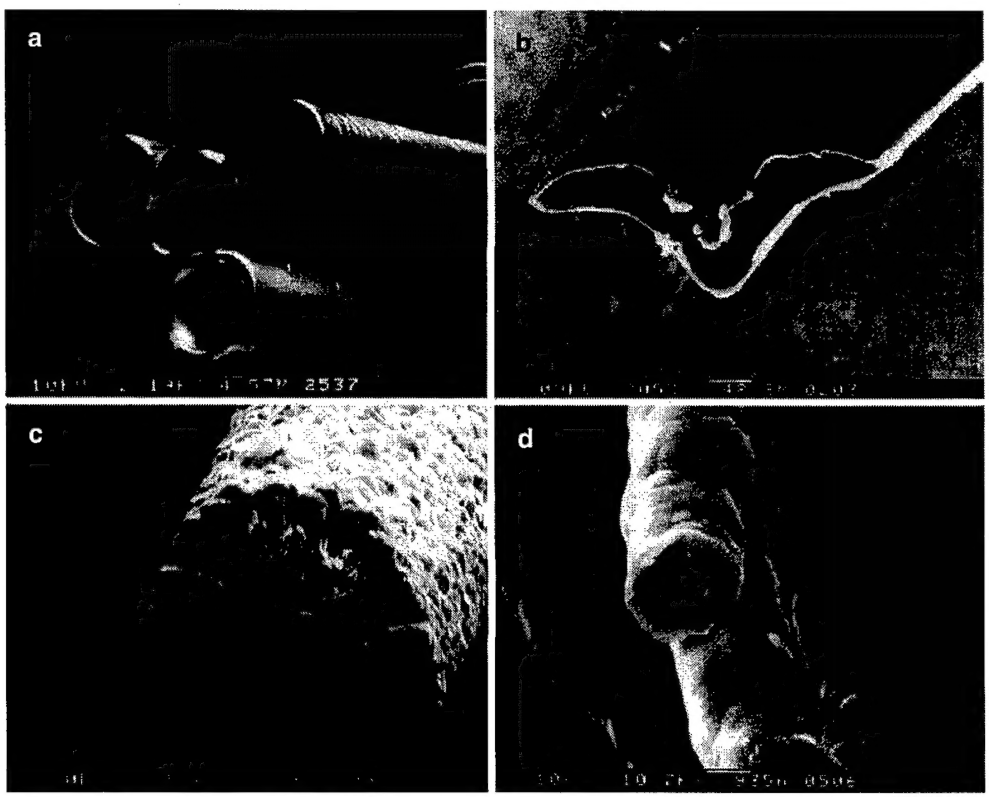


Fig. 1a–d Examples of microtubes. 10 micron silicon tubes (**a**) 410 micron nickel tubes (**b**) 26 micro silicon nitride tube (**c**) 0.6 micron quartz tube (**d**)

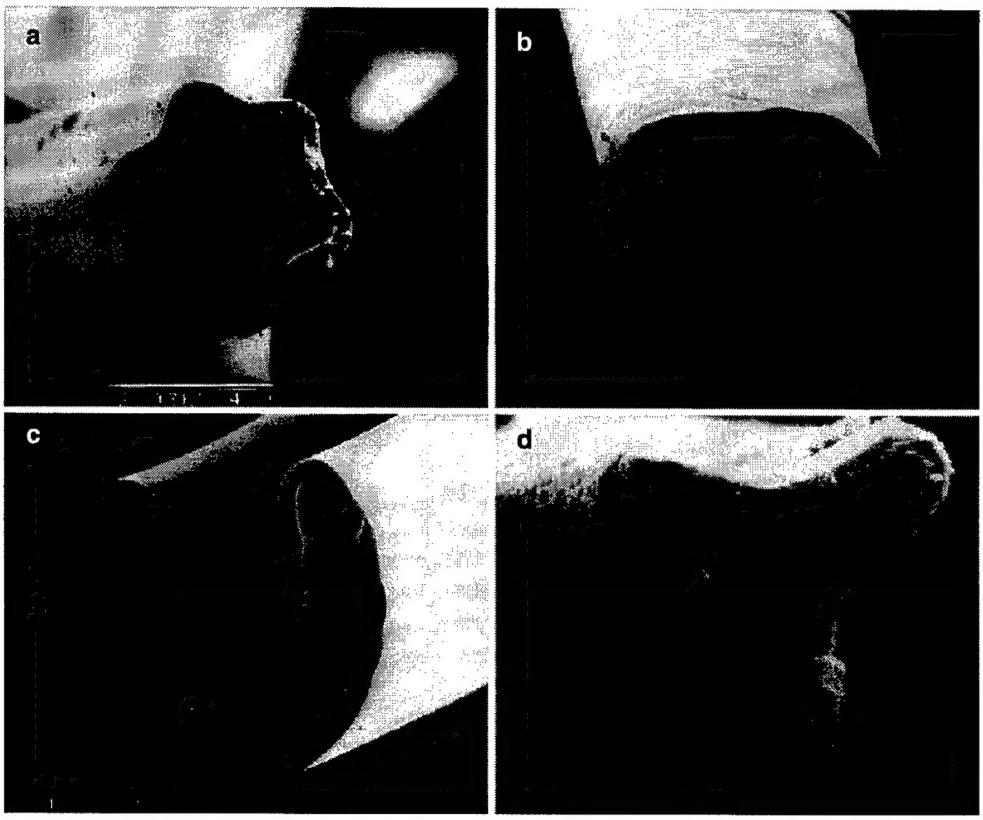


Fig. 2a–d Above 1 micron inside diameter, tubes can be made in any cross-sectional shape such as: 17 micron star (**a**) 9×34 micron oval (**b**) 59 micron smile (**c**) and a 45 micron trilobal shape (**d**)

(Fig. 3a). These thin-walled tubes are very useful for insulation or composite reinforcement. Thicker walled tubes (Fig. 3b), which are just as easily fabricated, are needed in some applications, such as those involving pressure. Microtubes tested to date have demonstrated surprising mechanical strength. In fact, preliminary studies with both copper and silver tubes with wall thicknesses in the micron range have shown that microtubes can have up to two times the tensile strength of an annealed wire with the same cross-sectional area of material.

In addition to free-standing microtubes, solid monolithic structures with micro-channels can be fabricated by making the tube walls so thick that the space between the tubes is filled (Fig. 4). The micro-channels can be randomly oriented or they can have a predetermined orientation. Any desired orientation or configuration of microtubes can be obtained by a fixturing process. Alternatively, composite materials can be made using a material different than the tube wall as a "matrix" that fills in the space between the tubes. The microtubes imbedded in these monolithic structures form oriented micro-channels which, like free-standing tubes, can contain solids, liquids and gases, as well as act as waveguides for all types of electromagnetic energy. If the tubes are placed in a solid structure they can act as lightweight structural reinforcement similar to that found in bone or wood. The cross-sectional shape of these reinforcement tubes can be tailored to maximize mechanical or other properties.

Besides being able to precisely control the tube wall thickness and composition, the interior surface of these tube walls can have practically any desired texture or degree of roughness. This control is highly advantageous and allows the use of microtubes in many diverse applications. For example, optical waveguides require very smooth walls while catalytic mixing-reactors would benefit from rough walls. (Because of the fabrication technique, the roughness on the interior of the tube wall can be quantified to a fraction of a micron). Furthermore, depending on the application, the walls of microtubes can range from non-porous to extremely porous as seen in Fig. 5. Porous walls would have application in chemical reactions and are useful in removing the mandrel.

Another unique feature of microtube technology is the ability to coat the interior or exterior surface of these tubes with a layer or numerous layers of other materials (Fig. 6). Alternating conductive and insulating layers would form a multiple-path conductor or a capacitor. A catalyst could be coated on the tube surface for chemical reactions. Other applications include using oxidation or corrosion protection layers on a structural tube material. In addition to layering, the tubes can be filled with another type of material to be used, for example, as a sensor or detector element.

Microtubes can be made straight or curved (Fig. 7), or they can be coiled (Fig. 8). Coiled tubes as small as 20 microns can be used, for example, as flexible connectors, or solenoid coils. For this later application, the coils could be of metal or of a high temperature superconductor with liquid nitrogen flowing through the tube. Another application for coils is for force or pressure measurement. No longer are we limited to quartz microsprings. With microtube technology, the diameter and wall thick-

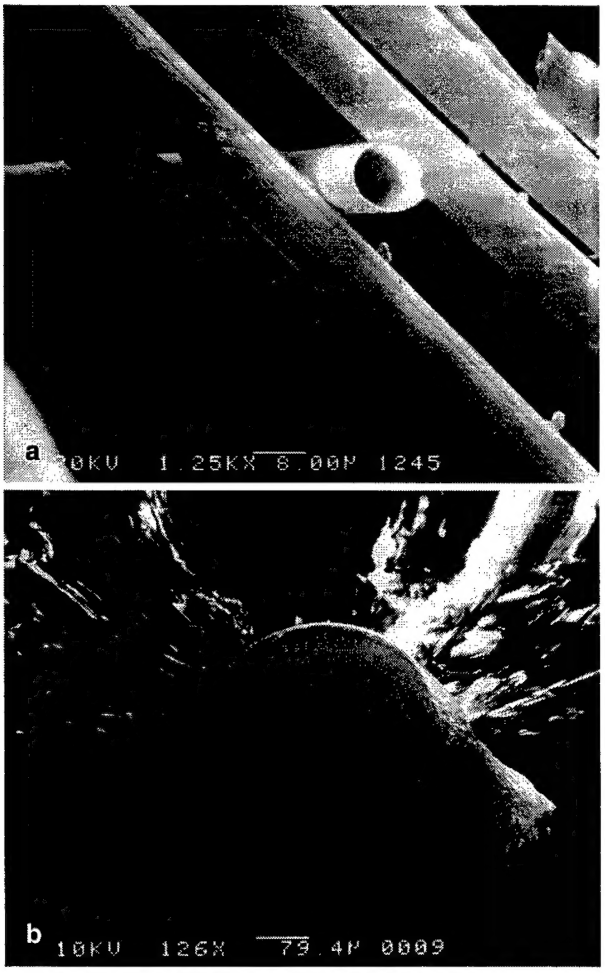


Fig. 3a, b Tubes can be structurally sound with very thin walls (**a**) or with thick walls (**b**)

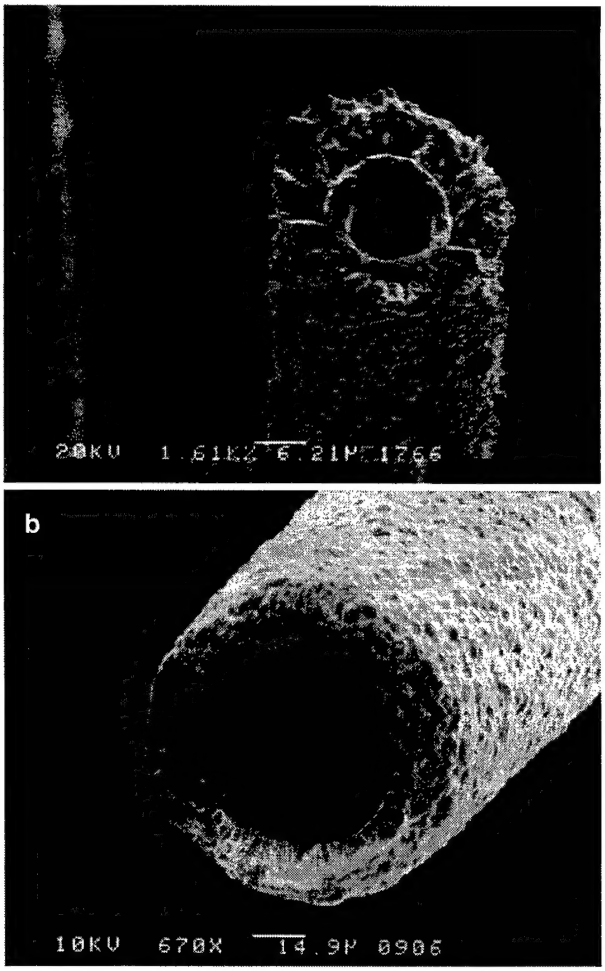


Fig. 6a, b Sapphire tube with silver liner. (**a**) Nickel tube with a silver liner (**b**)

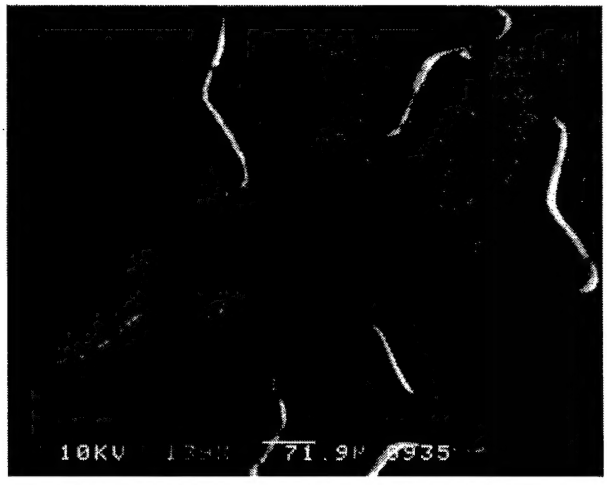


Fig. 4 Solid nickel structure with oriented micro-channels

ness of the tube, the diameter of the coil, the tube material and the coil spacing can be very precisely controlled to give whatever spring constant is needed for the specific application. In addition, a coiled spring tube wrapped around a core tube (Fig. 9) can be used as a counter-flow heat exchanger or as a screw drive for micromachines. [For this application, the wrapped coil cross-section could be made rectangular.]

Like the coiled spring tubes, bellows can be used as micro-interconnects and can be made in practically any shape imaginable. Figure 10a shows a belows with circular cross-section while the bellows in Fig. 10b has a square cross-section with aligned bellows segments. The bellows in Fig. 10c is a square bellows with a twist. A slightly more complex bellows is shown in Fig. 10d. This is a tapered-square camera bellows with a sun shade to demonstrate a unique capability of this technology. These bellows can have various shaped ends for connections to systems for use as, for example, finned heat exchangers, hydraulic couplings for gas and liquid, or static mixers for multiple fluids. The bellows in Fig. 10e has a thicker transition region and dove-tail on the end for connection to a device machined on a silicon wafer. The female dove-tail to mate with this bellows is a commercially available trench design [37] on a silicon wafer and provides a way to attach the bellows to the wafer which with proper sealing can be pressurized.

If one end of the bellows is sealed, an entirely new group of applications become possible. That is, if a bel-

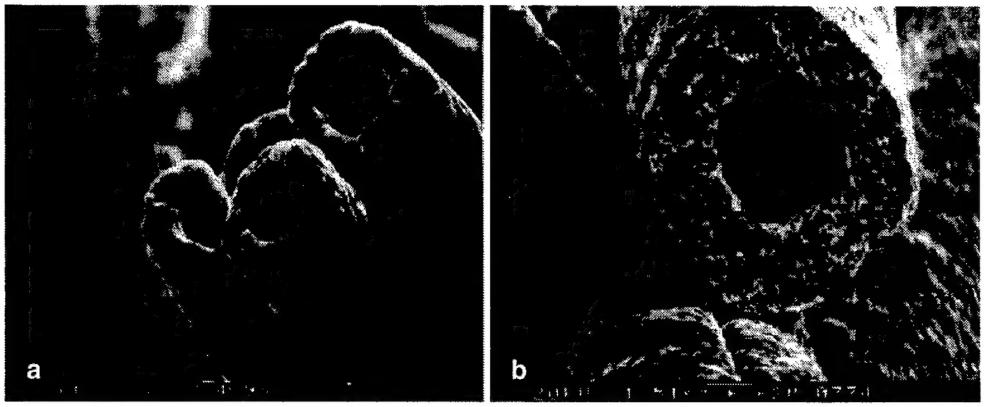


Fig. 5a, b Examples of porous tube walls. Pores mainly in the sides. (**a**) Pores mainly longitudinal (**b**)

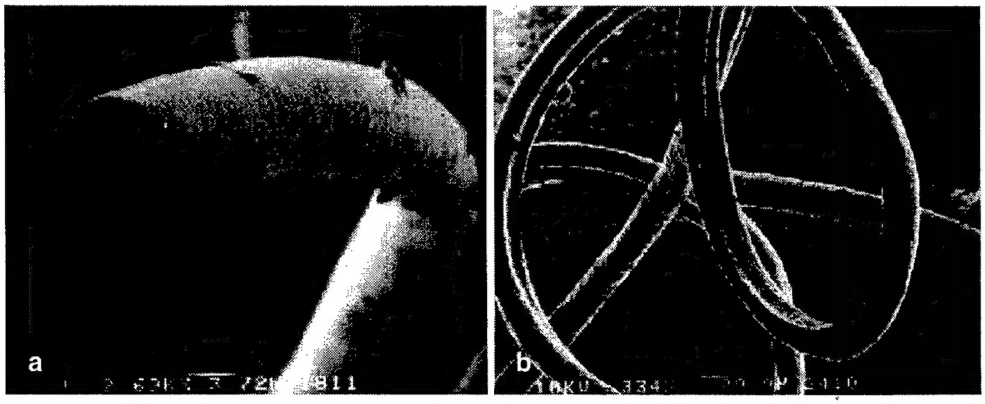
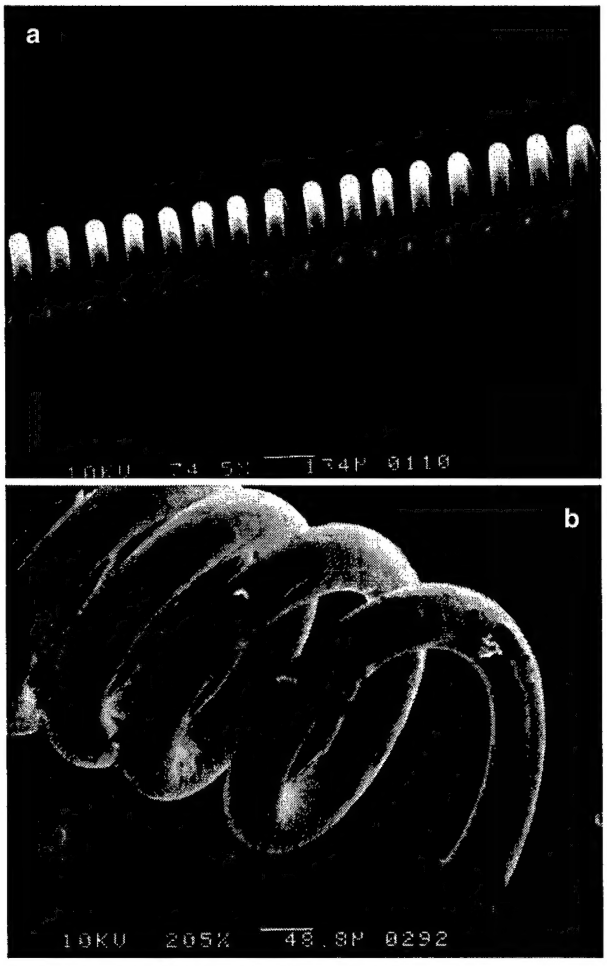


Fig. 7a, b Examples of a curved silver tubes. Single tube. (**a**) Multiple tubes (**b**)



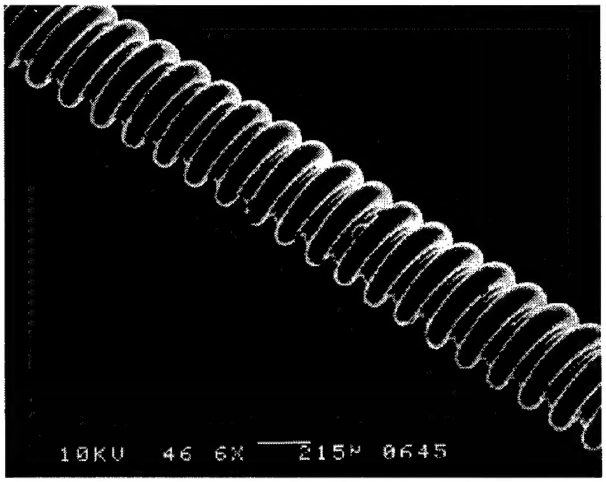


Fig. 9 A coiled tube wrapped around a tube or fiber that can be used as a heat exchanger or as a microscopic screw-drive

lows end is sealed, the bellows can be extended with hydraulic or pneumatic means, for example. In this configuration, a bellows could be used as a positive displacement pump, a valve actuator, or for micro-manipulation. As a manipulator, a single bellows could be used for linear motion, three bellows could be orthogonally placed for 3D-motion, or three bellows could be attached sever-

Fig. 8a, b Section of "large" coiled tube. (**a**) Open end of coiled tube (**b**)

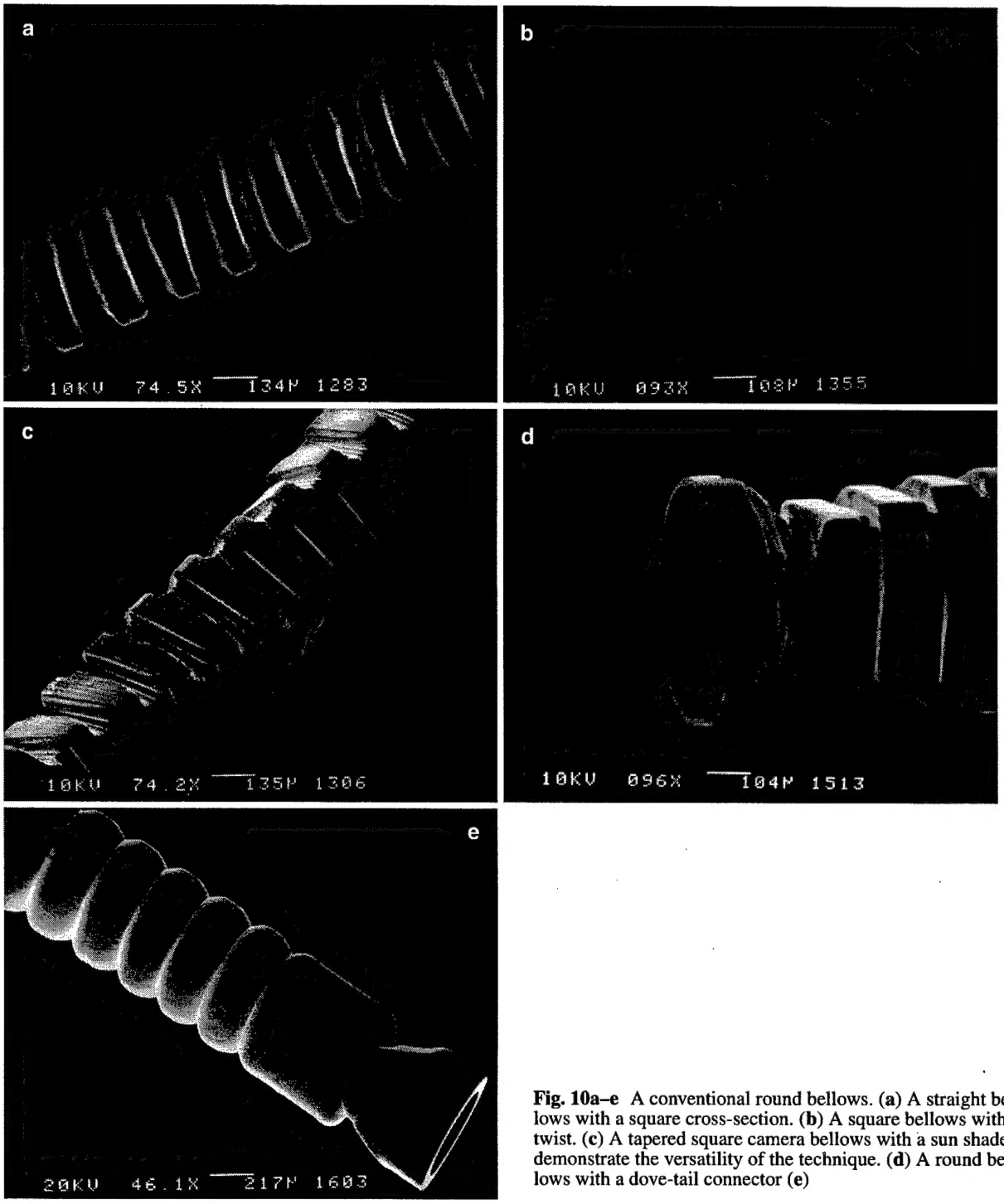


Fig. 10a–e A conventional round bellows. (**a**) A straight bellows with a square cross-section. (**b**) A square bellows with a twist. (**c**) A tapered square camera bellows with a sun shade to demonstrate the versatility of the technique. (**d**) A round bellows with a dove-tail connector (**e**)

al places externally along their axis and differentially pressurized to produce a bending motion. This bending motion would produce a micro-finger and several of these fingers would make up a hand. The large forces and displacements possible with this technique far surpass those currently possible by electrostatic or piezoelectric means and fulfill the need expressed by Wise and Najafi [6] when they stated that "In the area of micro-actuators, we badly need drive mechanisms capable of producing high force and high displacement simultaneously".

For most applications it is necessary to be able to interface microtubes with the macro-world. This is possible to achieve in a number of ways. For example, a tapering process can be used in which the diameter is gradually decreased to micron dimensions. Alternatively, the tubes can be interfaced to the macro-world by telescoping or through numerous types of manifolding schemes

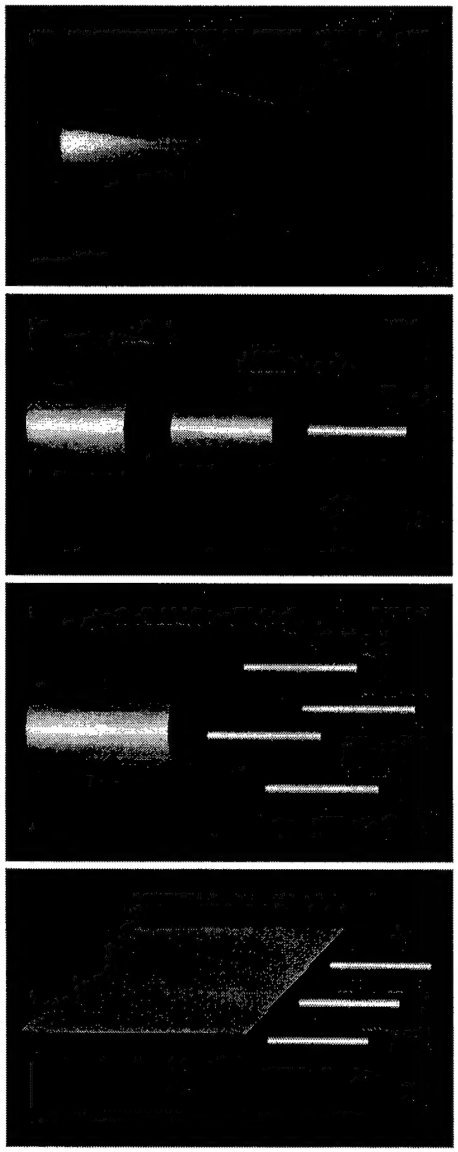


Fig. 11a–d Different ways of transitioning microtubes to the real world. Taper (**a**) telescope (**b**) bundle (**c**) manifold (**d**)

(Fig. 11). An example of a thin-walled 5 micron I.D. tube telescoped to a 250 micron O.D. tube can be seen in Fig. 12. A tube of this type could be used as a micro-pitot tube and, of course, could be made more robust by thickening the walls.

Although microtube technology has unique capabilities, it should be obvious that no single technology can fill all the requirements imposed by diverse applications. Thus, microtube technology can not easily compete with other technologies in certain applications. One of these involves, gas and liquid separation such as chromatography. For example, quartz tubing, which can be extruded and drawn in very long lengths, is inexpensive and available in micro dimensions. However, it should be noted that even in areas such as separation, there are niches for microtubes that involve the composition of the tube ma-

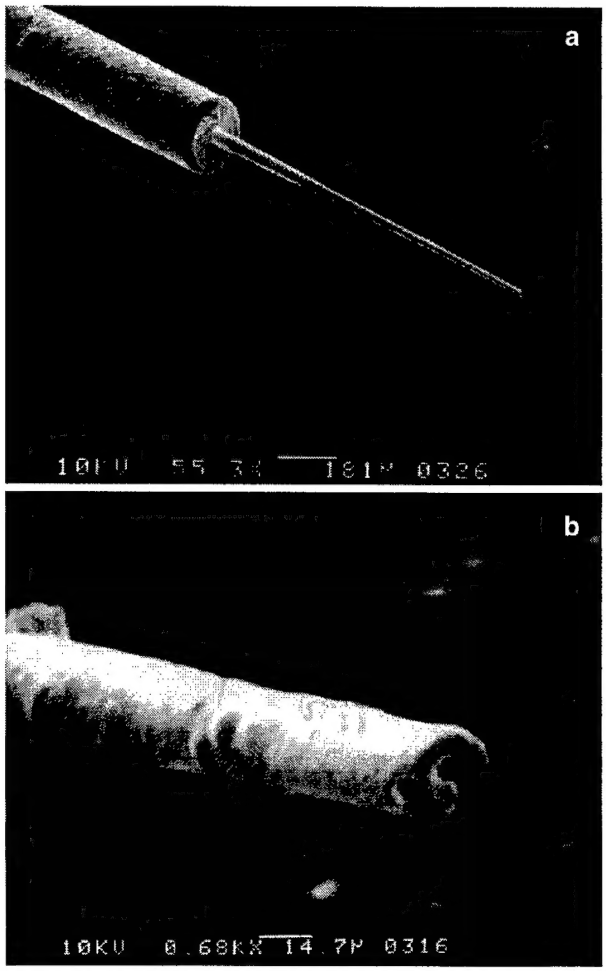


Fig. 12a, b A thin-walled 5 micron I.D. tube telescoped to a 250 micron O.D. tube. (**a**) View of the small open end of the telescope (**b**)

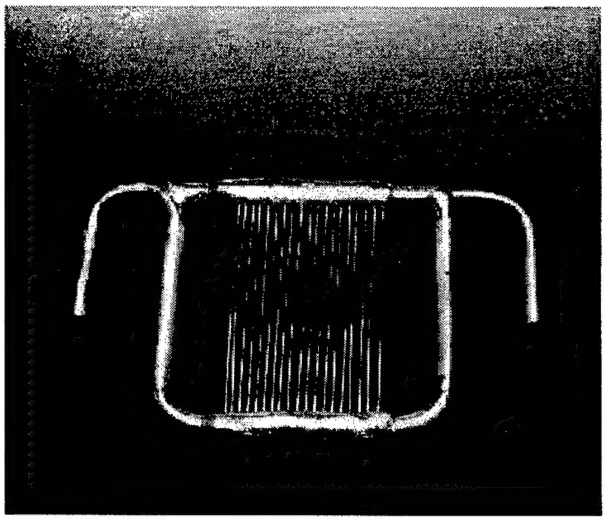


Fig. 13a, b Microtubes are manifolded to a tubular frame for gas separation

terial, the cross-sectional shape, or the inner wall coating. For example, Fig. 13 shows microtubes manifolded to a tubular frame for a specific gas separation application that requires microtubes of a specific composition with precise diameter and wall thickness.

Currently, these tubes have been made by a batch process in the laboratory, but the technique is equally suited to a continuous process which would not only be more efficient but in some cases much easier. Obviously, a continuous process would reduce costs. For most materials these are already rather low because, unlike some other processes, expensive tooling is not required. For many materials such as quartz, aluminum, copper, etc. the cost is anticipated to be much less than $ 0.01/cm. For precious metals such as gold or platinum, the cost would be significantly higher due to the cost of raw materials.

Conclusion

Microtubes appear to have almost universal application in areas as diverse as optics, electronics, medical technology, and microelectromechnical devices. Specific applications for microtubes are as diverse as chromatography, encapsulation, cross- and counter-flow heat exchange, injectors, micro-pipettes, dies, composite reinforcement, detectors, micropore filters, hollow insulation, displays, sensors, optical wave guides, flow control, pinpoint lubrication, micro-sponges, heat pipes, microprobes, plumbing for micromotors and refrigerators, etc. The technology works equally well for high and low temperature materials and appears feasible for all applications that have been conceive to date.

The advantage of microtube technology is that tubes can be fabricated inexpensively out of practically any material in a variety of cross-sectional and axial shapes in very precise diameters, compositions, and wall thicknesses orders of magnitude smaller than is now possible. In contrast to the other micro- and nano-tube technologies currently being developed, microtubes can be made out of a greater range of materials with a greater range of lengths and diameters, and with far greater control over the cross-sectional shape. These tubes will provide the opportunity to miniaturize (even to nanoscale dimensions) numerous products and devices that are currently in existence as well as allowing the fabrication of innovative new products that have to date been impossible to produce.

Acknowledgements The invaluable help Marietta Fernandez and Naomi Pilande in taking the SEM micrographs and of Peter Pollock in measuring mechanical properties is greatly appreciated. The financial support from Dr. Alex Pechenik of the Chemistry and Materials Science Directorate, Air Force Office of Scientific Research was responsible for much of this work.

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